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SPECIFIC HEATS OF AQUEOUS SOLUTIONS OF NaCl, NaBr, AND KCl.
COMPARISONS WITH RELATED THERMAL PROPERTIES

NAVAL WEAPONS SUPPORT CENTER
APPLIED SCIENCES DEPARTMENT
CRANE, IN 47522

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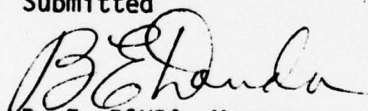FINAL REPORT for Period 1 March 1976 to 31 May 1977

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Submitted

B. E. DOUDA, Manager
Chemical Sciences Branch
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Applied Sciences Department

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Earlier unpublished measurements of the specific heat capacities of aqueous NaCl, KCl, and NaBr solutions from 5°C to 85°C, and from 0.05 $\underline{m}$ to saturation are presented. A twin calorimeter was used. A precision of nearly 1 part in 10^4 in the specific heat capacity is claimed. The results are compared with literature values (summaries or original data) for heat capacity, heat of dilution, and activity coefficient of these salts in solution by means of a polynomial in half-integer powers of molality and temperature. It is found that our values agree well with the more recent literature values of the heat capacities. Small systematic inconsistencies between the various types of data were found.

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PREFACE

The experimental work reported here was supervised by the late professor Frank T. Gucker, Jr. Lamb's work was supported by a Carnegie Post Doctoral Fellowship, and was performed in the Chemistry Department of Northwestern University, Evanston, IL. Tanner's work was supported by ONR Contract N6ori18007, Project No. NR051169, and was performed in the Chemistry Department of Indiana University. It was published as a masters thesis (1954), and is available from University Microfilms, Inc., Ann Arbor, MI, order number M-763. The computer calculations and write-up were performed at Naval Weapons Support Center, Crane, IN, under a grant from the Energy Research and Development Administration. The report has been submitted to the Journal of Solution Chemistry for publication.

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INTRODUCTION

Precision measurements of specific heat capacities and heats of dilution have been the subject of many investigations in this laboratory; and many improvements in experimental methods were developed (see reference 1 for a summary) in an attempt to get sufficient accuracy so as to be able to verify the Debye-Hückel theory. In this paper, we present heat capacity results obtained by the authors on binary aqueous solutions of NaCl, KCl, and NaBr. The values for NaBr fill a gap in the literature. The values for the other two salts significantly extend the concentration-temperature ranges, and in some areas improve on the precision of the available values, or confirm precise values already in the literature. A theoretical interpretation of the results is not attempted.

APPARATUS

The apparatus used was the one described in detail by Gucker, Ayres, and Rubin,[1] with minor modifications by the authors and by Dr. P. F. Van Velden while he was working in this laboratory.

Basically the apparatus consists of two similar calorimeters, one containing a solution, the other a fixed tare weight of water (about 270 g) enclosed side by side in a submarine jacket contained in an adiabatic water bath. Starting at the same initial temperature, both calorimeters (as well as the bath) are heated one degree by means of a pair of variable resistance coils connected in series. In several trials the resistance ratio is adjusted to find the ratio at which the

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two calorimeters would be heated at the same rate, as determined by a 20-junction thermel connected to a high sensitivity galvanometer. The specific heat capacity of the solution is then calculated from the measured resistance ratio and the known heat capacity of the calorimeters.

Corrections are applied (1) for the resistances of the leads to the calorimeter heaters (determined potentiometrically with a needle probe), and (2) for the differing amounts of heat required to maintain vapor saturation in the empty space at the top of the calorimeters.

The improvements performed since the writing of reference 1 were (1) the selection of Leeds and Northrup "Bureau of Standards" type resistors for the major elements of the measuring arms of the Wheatstone bridge used in the heating circuit, and the immersion of these in a thermostated oil bath, and (2) changes to the bath to allow rapid heating or cooling to the next starting temperature.

EXPERIMENTAL PROCEDURE

The calorimeters were calibrated by measuring the gross heat capacity ratio with varying amounts of water in one of them. A hollow copper shell was immersed to keep the water level the same. The values are 10.58 and 10.95 cal/deg, at 25°C, respectively, with a temperature coefficient of 0.29 cal/deg.

The resistance ratio of the measuring arms of the Wheatstone bridge, after calibration, is known to within 4 ppm. The resistance ratio required to produce equal heating rates is interpolated from galvanometer deflections from several trials using the nearest available resistance ratios. It is estimated to one part in 10^5.

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The actual scatter observed in repeated runs over the same temperature interval, without changing the liquid present in the calorimeters, indicates that the final calculated specific heat capacities are not known to better than one part in 10^4.

The sodium chloride used was reagent grade Baker and Adamson crystalline salt with a stated impurity content of no more than 0.02%. It was heated for several hours at 600°C, and stored thereafter in a dessicator. The potassium chloride and the sodium bromide were fused before use.

The salts and distilled water (not degassed) were weighed directly into the calorimeter, which was immediately suspended such as to seal off the open space at the top. In a few of the experiments with sodium chloride, measurements were performed with water in both calorimeters, then a small lid in one of them was opened briefly to insert a weighed chunk of the fused salt, and measurements were repeated over the same temperature interval. There was no difference in the results from the two procedures.

LITERATURE COMPARISON BY COMPUTER FIT

Our experimental results have been compared with relevant heat capacity, heat of dilution, and activity coefficient data from the literature by curve-fitting all to a common function of temperature and pressure, using a least-squares procedure. Existing summaries and correlations of literature data were used in place of the original data where possible.

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Silvester and Pitzer[2] have curve-fitted nearly all of the existing data on sodium chloride solutions at $t \geqslant 25°C$ to a 19-constant, theoretically derived function. To their tabulations of heat capacities thus smoothed we have added the values of smoothed heat capacities at 25°C tabulated by Parker,[3] and the heat capacities below 25°C of Perron, et. al.,[4] after making corrections as outlined by Desnoyers, et. al.[5]

Parker's[3] tables of smoothed heat capacities and relative enthalpies, and Hamer and Wu's[6] tables of smoothed activity coefficients were used to represent the literature for sodium bromide and potassium chloride solutions at 25°. At other temperatures original data were selected for comparison: references 7-10 (heat capacities, KCl), references 11-13 (heats of dilution, KCl), references 14 and 15 (activity coefficients, KCl), and reference 16 (activity coefficients, NaBr).

The coverage of activity coefficient data (EMF's, freezing and boiling points) was not intended to be inclusive. Papers were selected only where the activity coefficients had already been calculated by the respective authors. The results of Scatchard and Prentiss[17] were excluded because of the need to recalculate their activity coefficients using a better value of the cryoscopic constant.

The specific heat capacity data of Hess and Gramkee[18] for potassium chloride solutions were excluded because of obvious irregularities in their results.

In the references selected, all data presented between 0° and 100°C were included. Units were converted to international calories (4.184 joules) where necessary.

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As the basic function to which the data were fitted we have taken $\phi_H(\underline{m},\underline{t}) - \phi_H(0,25°) = f(\underline{m},\underline{t})$ to be a polynomial in half-integral powers of molality, $\underline{m}$, and Celsius temperature, $\underline{t}$. Here ϕ_H is the apparent molal enthalpy. The products of powers of $\underline{m}$ and $\underline{t}$ used here may be represented by the matrix

$$[1,\ \underline{t},\ \underline{t}^{3/2},\ \underline{t}^2,\ \underline{t}^{5/2}]^T \cdot [1,\ \underline{m}^{1/2},\ \underline{m},\ \underline{m}^{3/2},\ \underline{m}^2],$$

where superscript T indicates the transpose. Note that $t^{1/2}$ was omitted. Each matrix element is multiplied by a corresponding coefficient, $\underline{a}_{ij}$, obtained from a least squares procedure. The matrix for calculating the relative apparent molal enthalpy, ϕ_L, or the heat of dilution, ΔH_{dil}, would have zeros in the left-most column.

The matrix of $\underline{m}$ and $\underline{t}$ for the heat capacity, $\phi_{cp} = \partial f(\underline{m},\underline{t})/\partial \underline{t}$, corresponding to the same set of $\underline{a}_{ij}$, is then

$$[0,\ 1,\ 3/2\underline{t}^{1/2},\ 2\underline{t},\ 5/2\underline{t}^{3/2}]^T \cdot [1,\ \underline{m}^{1/2},\ \underline{m},\ \underline{m}^{3/2},\ \underline{m}^2].$$

The activity coefficient is given by $-2R\ \ln\gamma = \int \overline{L}/T^2 d\underline{t} + C(\underline{m})$, where $\overline{L} = f(\underline{m},\underline{t}) - f(0,\underline{t}) + \underline{m}\partial f(\underline{m},\underline{t})/\partial\underline{m}$ is the relative partial molal enthalpy, T (non superscript) is the Kelvin temperature and R is the molar gas constant. The functions of $\underline{m}$ and $\underline{t}$ corresponding to the common set of $\underline{a}_{ij}$ are no longer simple polynomials, but are represented by

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$$[\int 1/T^2 d\underline{t}, \int \underline{t}/T^2 d\underline{t}, \int \underline{t}^{3/2}/T^2 d\underline{t}, \int \underline{t}^2/T^2 d\underline{t}, \int \underline{t}^{5/2}/T^2 d\underline{t}, 1]^{\mathsf{T}} \cdot$$

$$[0, 3/2\underline{m}^{1/2}, 2\underline{m}, 5/2\underline{m}^{3/2}, 3\underline{m}^2].$$

The extra element of unity in the temperature vector represents the integration constant, $C(\underline{m})$.

Using all of the heat capacity, heat of dilution, and activity coefficient data simultaneously for solutions of a given salt, the coefficients $\underline{a}_{ij}$ are obtained. Smoothed values and deviations are then computed corresponding to each of the original data points.

A polynomial is much easier to use than a theoretically derived function, both for fitting the data here, and for later use in evaluating the thermodynamic functions at arbitrary temperatures and concentrations. The disadvantage is that a larger number of adjustable constants are required for a given degree of fit. In this work, the number of terms used did not quite allow a fit to within the experimental precision, although the systematic deviations of the heat capacities even in the worst cases are of the same order of magnitude as the experimental scatter.

Other simple functions were tried, but without improvement. A polynomial of the same number of terms, but only integral powers of Celsius temperature gave nearly as good a fit. A polynomial in Kelvin temperature was distinctly poorer. The use of the Debye-Hückel theory to determine the coefficients of $\underline{m}^{1/2}$, which are the $\underline{a}_{i2}$, gave a better fit at the lowest concentrations, but a poorer overall fit when the

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total number of terms was held fixed. This is as expected. Since the other terms had no theoretical significance, use of the Debye-Hückel constraint was abandoned so as to get the best fit with the fewest terms. However, in order to get good values of ϕ°_{cp} (ϕ_{cp} at infinite dilution) for the data of the present authors, these were fitted, each temperature separately, to a polynomial in powers of $m^{1/2}$, using the Debye-Hückel coefficients $\partial\phi_{cp}/\partial\sqrt{m} = 2/3\partial A_H/\partial t$ = 7.71, 8.09, 9.83, 11.57, and 13.09 cal/mole$^{3/2}$ deg at the temperatures 5°, 25°, 45°, 65°, and 85°C, respectively. These were obtained by differentiating values of A_H from reference 2.

In heat capacity or heat of dilution experiments, the quantities measured have a precision which is nearly independent of concentration, but they are then divided by the concentration to get the molal quantities ϕ_{cp} or ϕ_L. This suggests the use of a weight factor proportional to the square of the concentration. This was tried but resulted in systematically poor fits at low concentrations; a weight factor linearly proportional to concentration gave satisfactory results, and was applied to the KCl and NaBr heat capacity and heat of dilution data. Since the literature values for NaCl solutions were already smoothed sets,[2,3] no weights were used in fitting the NaCl data.

The heat of dilution experiments involve larger absolute quantities, and thus tend to make greater contributions to the least squares matrix. To compensate for this, all heat capacity data were arbitrarily weighted by an additional factor of x3, and the activity coefficient data by x50. In the latter case, this factor was still not large enough, as systematic

inconsistency between the activity coefficients and the other types of data manifested itself in systematically much poorer fits of the former (not presented here). This was shown to be not due to the type of smoothing function or its use--with a consistent set of thermodynamic values (the tabulations of reference 2) the degree of fit for the above polynomial to the activity coefficients alone was not lessened by the inclusion of enthalpy and heat capacity values.

RESULTS AND DISCUSSION

The experimental results of the present authors are presented as the apparent molal heat capacities in Tables 1-3 along with the values extrapolated to infinite dilution. The measured specific heats were converted to absolute heat capacities by the use of the values 1.00428, 0.99888, 0.99885, 1.00299, and 1.00387 for the heat capacity of pure water at 5°, 25°, 45°, 65°, and 85°C, respectively.[19]

These heat capacities and the selected literature values of heat capacities are compared in Figs. 1-4 by plotting the differences between the experimental values and smoothed values obtained by the procedure outlined earlier. In the case of Figs. 1 and 3, only heat capacity data were used in obtaining the smooth values, whereas in Figs. 2 and 4 the heat of dilution and activity coefficient data were also used.

Although a dozen or more temperatures may be involved, the points are grouped into ten-degree intervals centered above the temperatures used by the present authors. Data of the other authors falling outside these intervals were used in the least squares procedure, but are not

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represented in the plots. The keys associated with the figures give more precise information.

The coefficients, $\underline{a}_{ij}$, corresponding to the figures are given in Table 4. They should be useful for calculation of thermodynamic properties, especially specific heats and enthalpies, at arbitrary concentrations and temperatures. For activity coefficients, the results of such calculations would be less accurate, but still useful where experimental data are not available.

Considering the NaCl plot (Fig. 1), there is good agreement among the values of the present authors, and those of Perron, et. al.,[4,5] and the smoothed values of Parker.[3]

The smoothed values of Silvester and Pitzer[2] show large systematic deviations from the others as one approaches 25°, the cutoff temperature below which they state they cannot fit the literature data with the theory they have thus far derived.

Considering KCl solutions (Figs. 2 and 3), the values of the present authors are in good agreement with the recent results of Rueterjans, et. al.,[8] except at the highest temperature, where there appears to be systematic disagreement among all three sets of data involved.[7,8] There is also a small systematic disagreement with the values of Parker[3] at 25°C. The older values of references 9 and 10 were omitted from the calculations for Fig. 3, so as to get the best possible set of coefficients.

The larger systematic deviations from the smoothed functions in Fig. 2, compared to Fig. 3 are taken as evidence of some mutual inconsistency of heat capacity, heat of dilution, and activity coefficient data, as discussed earlier.

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Regarding NaBr solutions (Fig. 4), our values are in good agreement with the rather limited data in the literature, as represented by Parkers[3] tables of smoothed values at 25°. The scale was expanded enough to show the roundoff errors of these latter. There was no chance to test the consistency of the enthalpy values[3] with the others since the former were available only at one temperature, 25°C. A considerable inconsistency between heat capacities and activity coefficients manifested itself in the deviations of the experimental activity coefficients[6,16] from the smooth values (plot not shown).
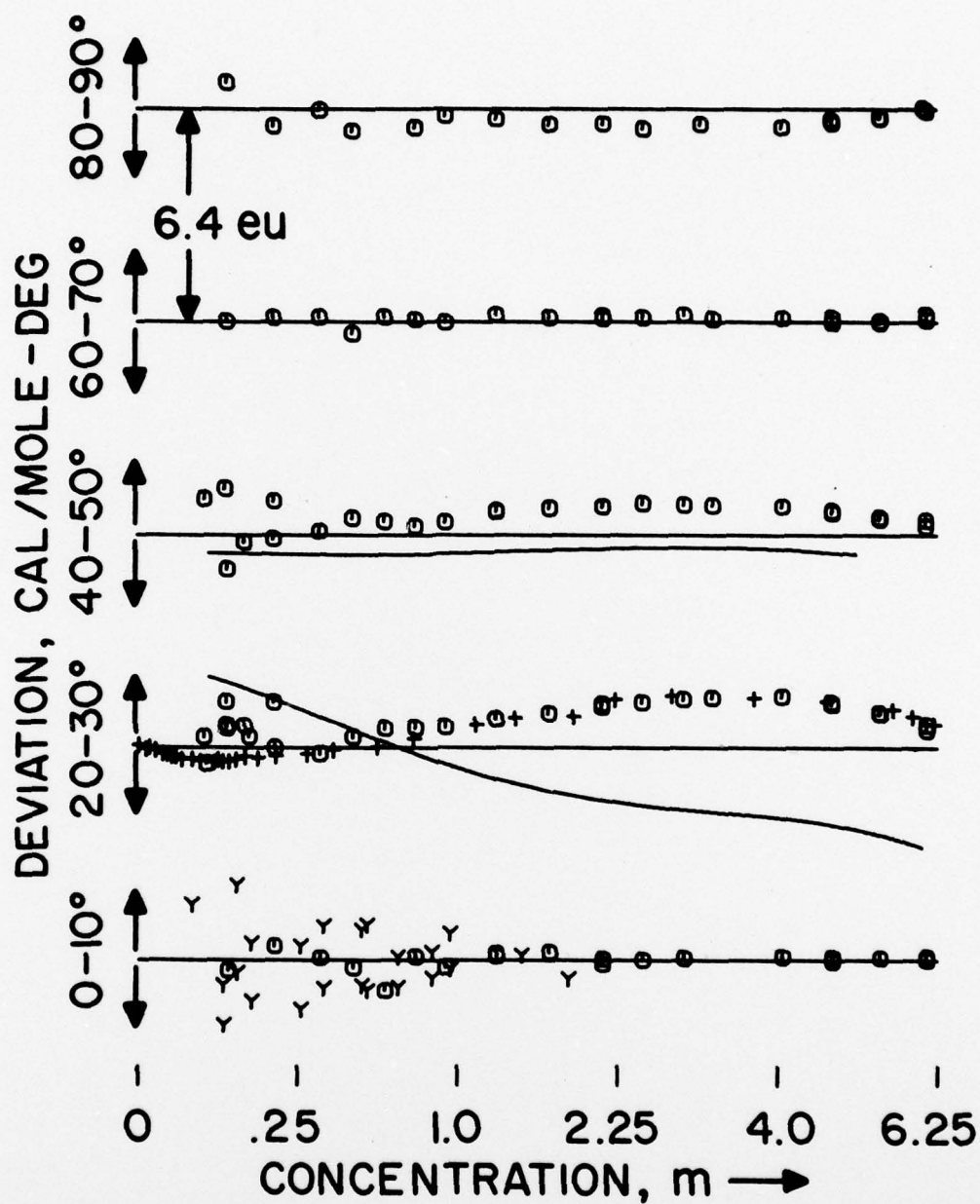
FIG. 1. NaCl Solutions. Deviations of experimental apparent molal heat capacities from smoothed values. The symbols are: □, this work; Y, reference 4,5; +, reference 3; —, reference 2.

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FIG. 2. KCl Solutions. Deviations of experimental apparent molal heat capacities from smoothed function fitting the heat capacity, heat of dilution,[3,11-13] and activity coefficient[6,14,15] data.

Reference	This work	7	8	9	10	3
Symbol	□	X	+	Z	Y	—
Group (from top)	Experimental temperature, degrees Celsius					
5	85	80	80,90		86.5	
4	65		60,70			
3	45		40,50	40	39.2	
2	25		30	20,25,30	23	25
1	5			10		

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Figure 2

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FIG. 3. KCl Solutions. Deviations of experimental apparent molal heat capacities from smooth function fitting only the heat capacity data of reference 3, 7, 8, and this work. The symbols are as in Fig. 2.
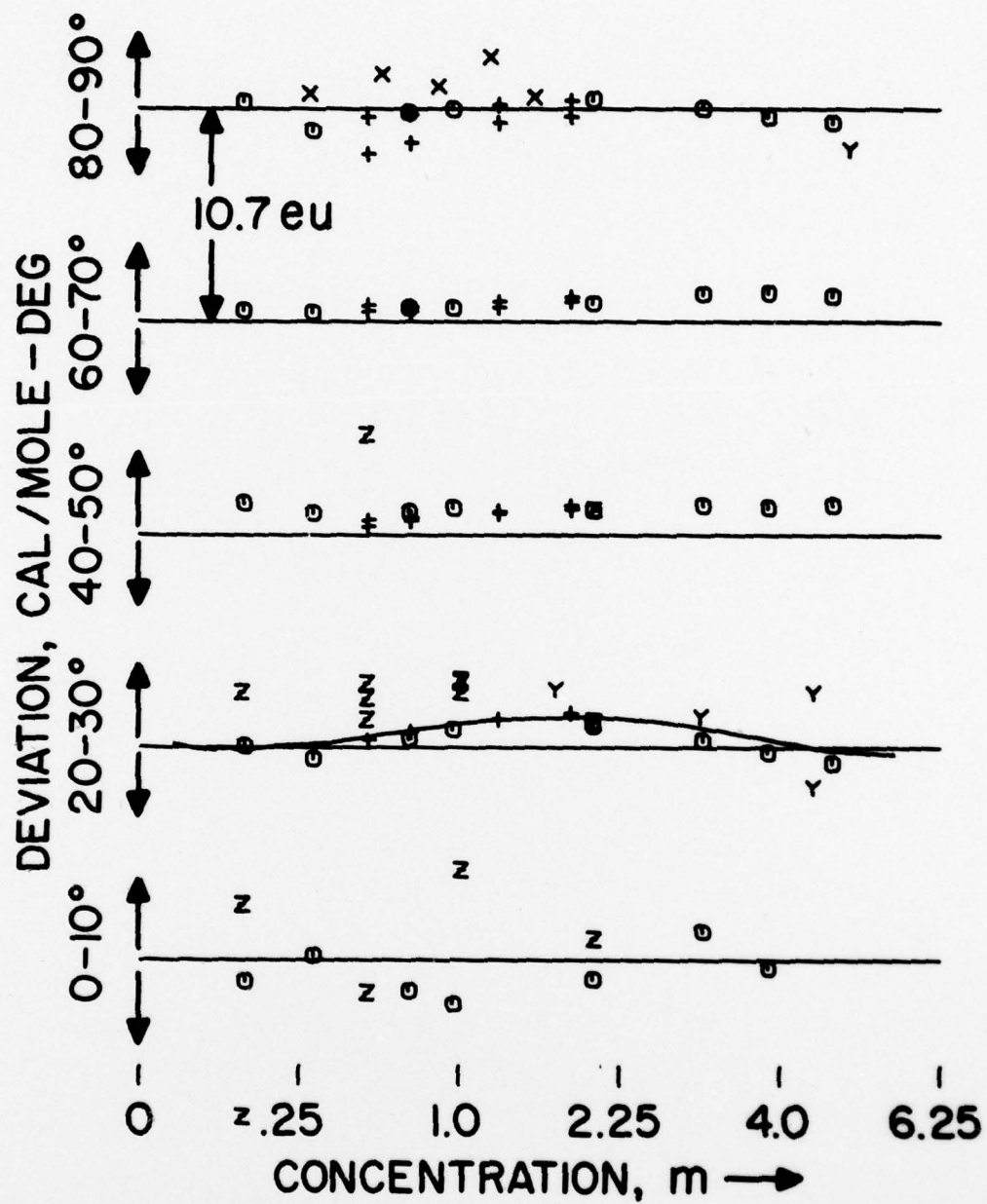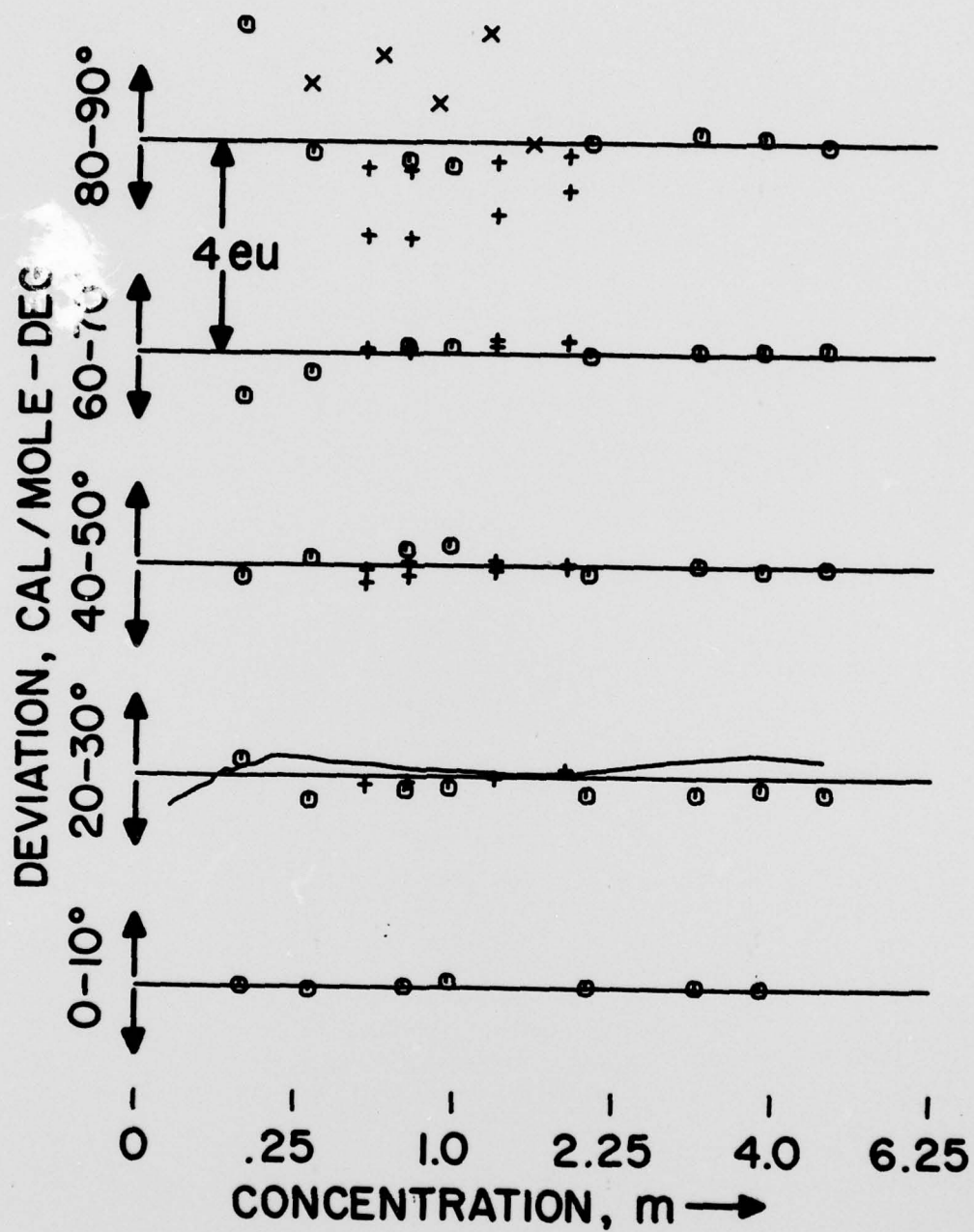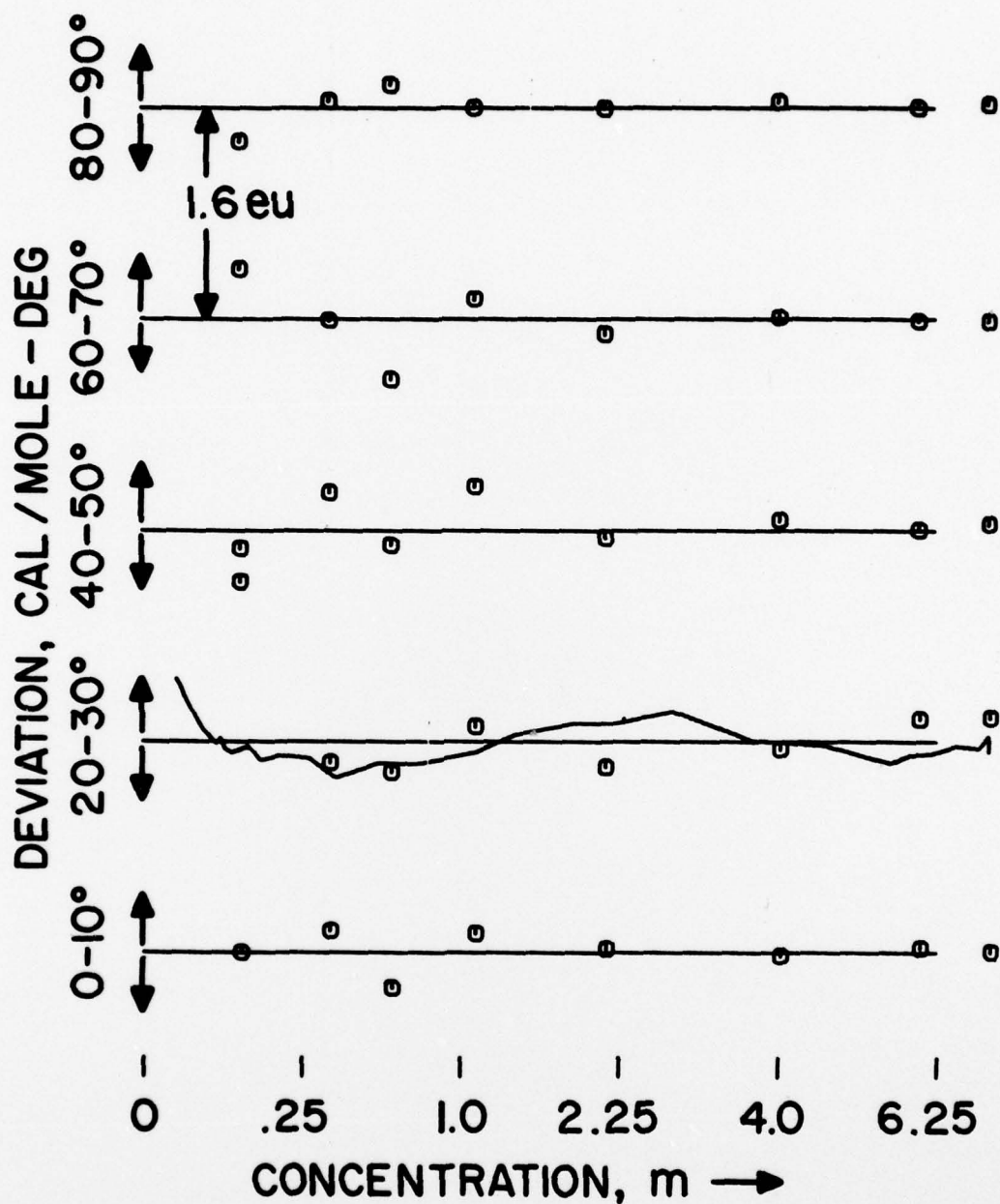
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FIG. 4. NaBr Solutions. Deviations of experimental apparent molal heat capacities from smooth function fitting all of the heat capacity, heat of dilution, and activity coefficient data. The symbols are: □, this work; —, reference 3.

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Table 1. Apparent Molal Heat Capacities of NaCl Solutions, Experimental and Extrpolated to Infinite Dilution.

$\phi_{cp}, \phi^{\circ}_{cp}$ int. cal/mole-deg

Molality	5°C	25°C	45°C	65°C	85°C
0.0	-38.0	-21.1	-15.5	-14.3	-15.4
.0411		-19.38			
.0424			-12.69		
.0450		-20.08			
.0740			-11.79		
.0748		-18.38			
.0754		-17.58			
.0763	-34.65	-18.28	-14.18	-11.33	-11.34
.1101		-17.68	-12.89		
.1177		-17.88			
.1782			-11.99		
.1798	-31.66	-15.94	-10.84	-10.03	-11.39
.1814		-17.28			
.3211	-29.34	-15.80	-10.48	-8.90	-9.70
.4480	-27.50	-14.05	-9.18	-8.58	-9.44
.5918	-25.97	-12.55	-8.37	-7.26	
.7493	-22.72	-11.30	-7.63	-6.54	-7.57
.9197	-20.84	-10.09	-6.64	-5.84	-6.36
1.2460	-16.78	-7.83	-4.82		
1.2461	-16.69	-7.83	-4.79	-4.22	-4.99
1.6457	-12.76	-5.50	-3.07	-2.79	-3.55
2.0975	-9.23	-3.17	-1.31	-1.12	-1.92
2.0979	-9.40	-3.06	-1.31	-1.21	
2.4722	-6.61	-1.41	.10	.05	-.84
2.8978	-3.92	.36	1.52	1.47	
3.0743					1.07
3.2114		1.58	2.49	2.27	
4.0286					3.44
4.0338	1.71	4.49	4.95	4.56	
4.6661					5.07
4.6873	4.26	6.40	6.61	6.04	5.02
4.6877	4.14	6.45	6.66	6.19	
5.3440		8.22	8.18	7.52	6.45
5.3572	6.58	8.27	8.19	7.52	6.46
5.3573		8.26	8.23	7.55	6.48
5.9883					7.81
6.0438	8.79	10.09	9.85	9.11	
6.0439	8.68	9.95	9.69	8.94	7.82

Table 2. Apparent Molal Heat Capacities of KCl Solutions, Experimental and Extrapolated to Infinite Dilution.

ϕ, ϕ_{cp} int. cal/mole-deg

m	5°C	25°C	45°C	65°C	85°C
0.0	-44.4	-28.0	-23.4	-23.3	-22.0
.1058	-40.47	-24.97	-20.38	-19.86	-18.27
.2918	-36.50	-23.98	-19.16	-18.18	-18.02
.7036	-30.82	-20.65	-16.79	-15.56	-15.45
.9546	-28.14	-19.01	-15.48	-14.49	-14.54
1.4942	-20.33	-14.16	-11.93	-11.08	-10.96
3.0789	-14.69	-10.54	-8.73	-8.18	-8.09
3.8334	-11.89	-8.45	-7.14	-6.56	-6.45
4.6618		-6.56	-5.42	-4.97	-4.92

ϕ_{cp}, ϕ°_{cp} int. cal/mole-deg

Molality	5°C	25°C	45°C	65°C	85°C
0.0	-42.2	-22.0	-17.3	-15.5	-17.3
.0942	-34.36	-18.51	-14.33	-12.14	-13.75
.3457	-31.45	-16.68	-11.03	-9.81	-10.48
.6112	-27.00	-13.93	-9.41	-8.51	-8.51
1.0453	-19.84	-9.63	-6.17	-5.56	-6.31
2.1224	-10.01	-3.82	-2.10	-2.09	-2.69
4.0188	1.32	3.90	4.19	3.49	2.47
5.9724	6.69	9.58	9.02	8.06	6.77
7.5903	13.06	13.23	12.36	11.18	9.83

Table 4. Solutions, a_{ij}, from Least Squares Fit

(a) NaCl (Fig. 1)

i/j	1	2	3	4	5
2	-.5032065E2	-.9524834E1	.4564850E2	-.1695425E2	.1593134E1
3	.4061186E1	.4436629E1	-.6684396E1	.1093451E1	.2502421E0
4	.6781517E-1	.3723844E0	.9963251E-1	.2506304E0	-.9363895E-1
5	-.1621504E-1	.1014934E-1	.1503233E-1	-.2034605E-1	.5413035E-2

(b) KCl, all data (Fig. 2)

i/j	1	2	3	4	5
1	.7363226E3	.1188364E4	-.2502991E4	.1430572E4	-.29482176E3
2	-.2225701E2	-.2887327E3	.5928382E3	-.3978245E3	.86911624E2
3	-.4345848E1	.9637115E2	-.1932975E3	.1305238E3	-.28406550E2
4	.7603808E0	-.1133012E2	.2272248E2	-.1536835E2	.33300047E1
5	-.3580932E-1	.4606441E0	-.9211753E0	.6221033E0	-.13411598E0
6		-.3280868E5	.6571001E5	-.4446519E5	.96300747E4

(c) KCl, recent Cp data (Fig. 3)

i/j	1	2	3	4	5
2	-.6207288E2	.1271225E2	.7385058E1	.3135719E1	-.2138526E1
3	.3497589E1	.8305989E1	-.8735840E1	.1446443E1	.3143065E0
4	.5069190E0	-.2587351E1	.2504271E1	-.8133720E0	.6548959E-1
5	-.5054430E-1	.1715447E0	-.1686952E0	.6306781E-1	-.7506554E-2

(d) NaBr (Fig. 4)

i/j	1	2	3	4	5
1	.9000258E3	.2377693E2	-.7821250E3	.2333850E3	-.1690882E2
2	-.7086105E2	.3937498E2	-.3269203E1	.9984035E0	-.3693087E0
3	.8998691E1	-.8825127E1	.7002866E1	-.3330009E1	.5561056E0
4	-.3960249E0	.9199868E0	-.1205143E1	.6050396E0	-.9812187E-1
5	-.1862440E-2	-.3037965E-1	.5395432E-1	-.2827960E-1	.4603164E-2
6		.2561783E4	-.3675935E4	.1848771E4	-.2983654E3

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